RADIATE
Ion Implantation
Quality Assurance Manual

ISSUE STATUS

Issue	Amendment/Comment	Author	Approved
1	Creation of amalgamated Implant QA documents from partners	RPW (Dec 2019)	
1.1	Correction of typographical errors and improvemnets generally of the text	CC (Dec 2019)	

Contents

1. Introduction

This document covers the quality assurance procedures at the implantation facilities providing Transnational Access as part of the RADIATE consortium.

RADIATE is an INFRAIA (Integrating and opening research infrastructures of European interest) project in H2020 with the main objective to provide user access to Europe's key ion beam facilities. More than 15.600 hours in total of trans-national access are offered to users at 11 ion beam facilities in Europe during the project duration of 4 years. The portfolio of the ion beam facilities ranges from specialized low-energy ion beam systems to large accelerators far beyond laboratory scale. They are operated as user facilities offering access to very specialized analysis and materials modification techniques for users from academia and industry. The facilities offer ion beam services in two application areas: (i) ion beam analysis; and (ii) ion beam modification.

This manual specifically covers ion implantation and modification. A separate manual covers ion beam analysis. Bespoke implantation experiments and analysis may also require their own separate Quality Assurance (QA) procedures on an "as required" basis.

Ion beam modification includes ion implantation for low ion fluences and ion irradiation for high ion fluences, respectively. Implantation and irradiation are done homogeneously on a large area with a broad beam or locally with a focused beam and with single ion impacts. The depth of the modification or implantation can be adjusted between several hundreds of micrometres by using swift heavy ions with energies between few MeV and GeV and a few nanometres by using low-energy or highly-charged ions. In particular for surface modifications and implantation in 2D materials ultra-low ion energies of a few tens of eV can be provided.

RADIATE QA meetings take place twice per year via video conference. The results of QA testing must be reported at these meeting and any proposed adjustments to the QA procedures should be discussed there prior to implementation.

The following text outlines the QA procedures and their justification for implantation. They have been formally summarised in the literature [i].

2. Ion Implantation QA

Traditionally the main concerns with ion implanters are the accuracy of the implant fluence, uniformity and energy. Such criteria can be influenced by many experimental variables, therefore to routinely examine these machines it is imperative that the experimental test parameters are firmly set and repeated regularly.

The procedures in this section are based on procedures adopted at the Surrey Ion Beam Centre for many years and should be used at other TNA providers engaged in implantation (as opposed to irradiation) service in the absence of any procedures of their own.

2.1 Ion Implantation QA test material

Silicon wafers are used for quality assurance purposes. QA wafers have the following specification:

a) Growth method: Cz
b) Grade: PRIME
c) Diameter 100mm
d) Thickness 525 ± 25um
e) Orientation <100>
f) Finish - polished on one side
g) Resistivity between 1-10Ω-cm
h) Type/dopant: either n/phosphorus or p/boron
i) A single primary flat, no secondary flat

If other material is used it should be recorded appropriately and used consistently in future QA runs to provide useful comparisons.

2.2 Ion Implantation QA schedule

With careful planning, a quarterly inspection provides enough information and traceability of ion implantation over the year to highlight any potential problems.

Each Quarter, QA wafers are implanted to check implant repeatability, implant species dose sensitivity and absolute dose (properly, fluence). The choice of implant energies and doses are specific to what each machine can feasibly provide from within their respective energy windows. Currently the following routine implants are performed each quarter to check wafer-to-wafer implant repeatability.

Medium Energy Implanter 30 keV Boron, fluence = $1x10^{15}$ cm^{-2}

Medium Energy Implanter 150 keV Arsenic, fluence = $5x10^{14}$ cm^{-2} (10^{15} cm^{-2} for RBS)

High Energy Implanter 500 keV Antimony, fluence $2x10^{14}$ cm^{-2} ($1x10^{15}$ cm^{-2} for RBS)

A minimum of five wafers are implanted each quarter. Of these wafers a minimum of three are annealed and analysed immediately. The other wafers are stored and then one is annealed and measured alongside the wafers from each of the next two quarters. These tracer wafers then make it possible to rule out any processing issues from quarter to quarter.

For those implantation facilities without an annealing and electrical capability should forward QA wafers to Surrey for annealing and electrical measurement. It is expected that they will be able to do their own in-house RBS measurements.

The number of wafers and their intended purpose are clearly indicated in the list. For the wafers marked for four-point-probe (4pp) mapping then wafers need to be annealed to remove the implant damage and electrically activate the implanted dopants. Typically a 1000ºC or a 950ºC anneal for duration of 3 minutes is sufficient to electrically activate the Boron, Arsenic and Antimony implanted wafers as the doses are well below the solid solubility at these temperatures.

The wafers marked for Rutherford Backscattering Spectrometry (RBS) are set aside for the total implant fluence to be quantified, not only in the centre of the wafer but also towards the extremities of the wafer to obtain quantification of spatial non-uniformity.

The general methodology behind the QA schedule has been explained. To gain further insight to the QA procedures it is necessary to explain and define the experimental processes which follow the implantation stage.

Taking into account implantation and analysis time table 1, below, presents the quarterly implantation matrix for quality assurance. The doses and energies have been specifically tailored to ensure the detection of possible implantation errors whilst being independent of any possible annealing problems.

Medium Energy Implantation **Std QA – B and As Repeatability**	**High Energy Implantation** **Std QA – Sb Repeatability**
6 x B 30keV $1x10^{15}$ cm^{-2} (4x4pp, 2xStore)	5 x Sb 500keV $2x10^{14}$ cm^{-2} (3x4pp, 2xStore) 3 x Sb 500keV $1x10^{15}$ cm^{-2} (2xRBS, 1x4pp)

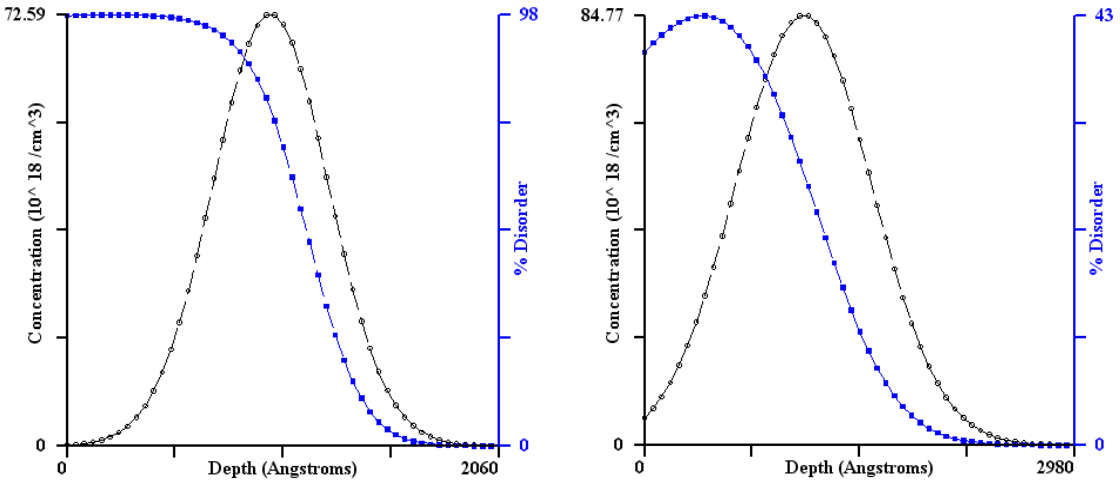
6 x As 150keV 5x10^{14} cm^{-2} (4x4pp, 2xStore) 3 x As 150keV 1x10^{15} cm^{-2} (2xRBS, 1x4pp)	

Table 1: Schedule for Implantation each QA period; all implants are performed at 7˚ tilt and 22˚ rotation to avoid channelling.

3. Annealing

The thermal treatment prior to the electrical analysis of the QA wafers is a crucial step which must be completed with enough of a thermal budget to achieve 100% electrical activity whilst removing the damage sustained to the substrate, therefore creating layers which are sensitive to the implant dose and not the thermal processing.

For Boron and Arsenic in silicon the solid solubility is greater than 10^{20} cm^{-3} at 1000ºC, for the majority of the doses are therefore chosen to be lower than this value. Figure 1 illustrates the implant and disorder distributions for the QA implants of As and B studies. Figure 2 illustrations the rapid thermal anneal profile (temperature versus time) for the QA Boron implanted wafers. These wafers are annealed at 1000ºC for 3 minutes as illustrated in figure 2. The As and Sb QA implanted wafers are annealed at 950ºC for 3 minutes.



As 150keV – 5x10^{14} cm^{-2} B 30keV – 1x10^{15} cm^{-2}

Figure 1: Implant and disorder distributions for the As and B QA implants.

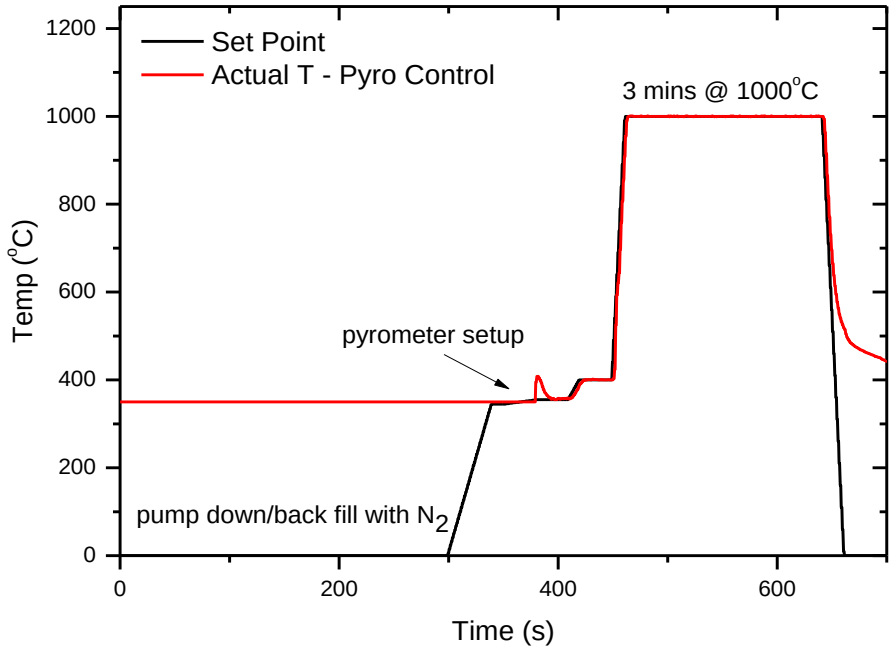


Figure 2: A typical rapid thermal anneal profile for a Boron implanted QA wafer.

4. Four-point-probe (4pp) Wafer Mapping

The 4D 4pp wafer mapper should be used to examine the spatial uniformity of the implanted wafer via simple sheet resistance (Rs) measurements. The principle of the technique is out of the scope of this document but the following information will aid in determining when the output data is good and give an idea of the repeatability and accuracy of the technique.

With any experimental process it is always advisable to check the functionality of the machine before measuring the QA wafers. Wafers should be kept clean by handling with plastic wafer tweezers or vacuum wand (if available). Figure 3 shows an example of a sheet resistance map of a 6” silicon wafer implanted with Boron under the indicated conditions.

The wafer sheet resistance is measured at each location marked by a cross. The contour maps are then computer generated. For 6” diameter wafers the sheet resistance is measured using a 10 mm grid pattern. For 4” wafers the measurements are made at 5 mm intervals across the wafer. Measurements are not taken within 10 mm of the wafer edge in order to eliminate wafer geometry effects on the sheet resistance measurement. It is good practise to make a repeat measurement on each wafer. A complete wafer map typically takes only a few minutes to complete the measurement.

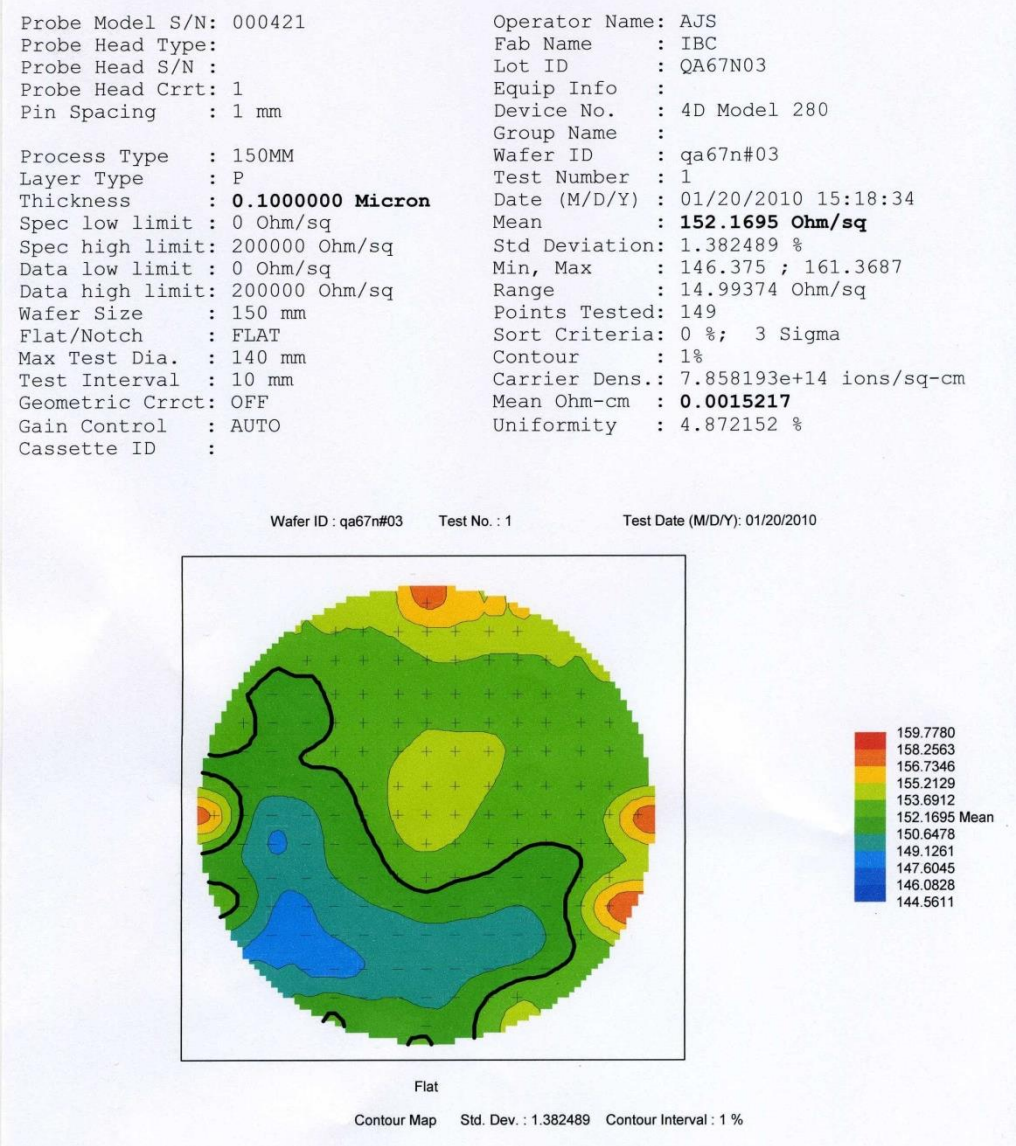


Figure 3: A typical sheet resistance map by the 4D four point probe machine of a Boron implanted 6"wafer number- QA67N#03.

Figure 4 shows a sheet resistance map of a non-uniform implant across a 6" wafer. Figure 4 illustrates a red ring, indicative of a high resistance region around the edge of the wafer which is due to the implanted area being much smaller than the wafer itself due to implanting through a 4" diameter aperture.

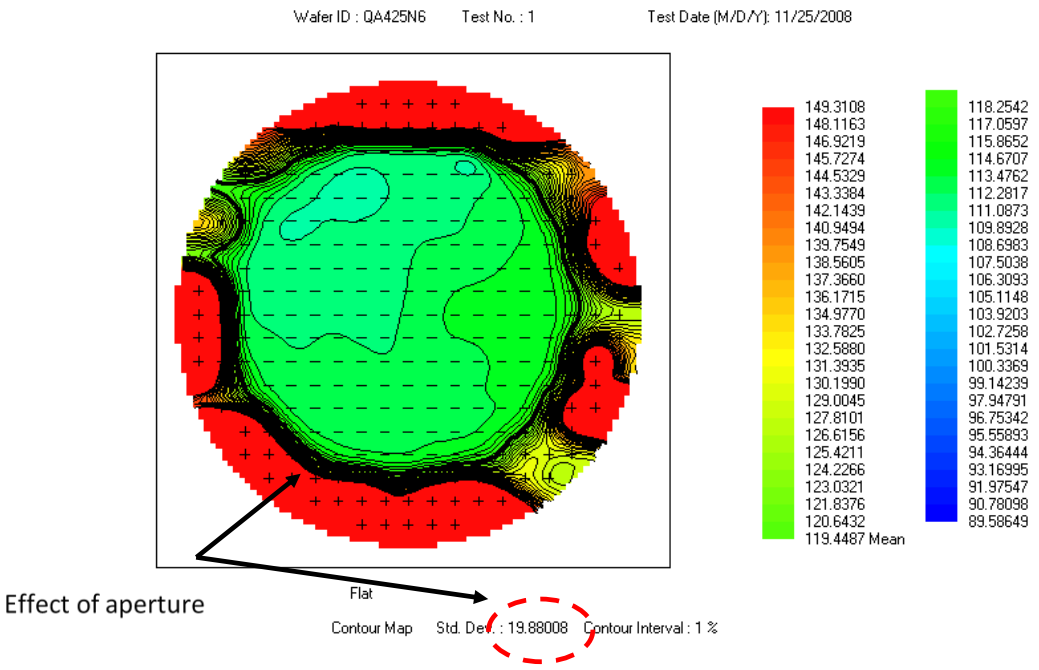


Figure 4: shows a sheet resistance map of a non-uniform implant

4.1 Probe Repeatability

It is possible to check the 4pp head by using a built-in diagnostic program which is a probe head repeatability test. Table 2 presents data from 5 successive measurements from 4 unique specifiable locations using this program. The table indicating that at all 4 measurement sites the repeatability is below 0.5%. If the repeatability is greater than 0.5% the probe head should be cleaned and roughened slightly by making repeated single point measurements on a special ceramic sample supplied with the instrument.

4.2 4pp Mapping – Potential Errors

The 4pp head relies on penetrating any surface oxide that may be present and making good ohmic contacts on all 4 probes to the surface of the substrate in question. The head is weighted in such a manner to penetrate a surface oxide in the order of a native oxide (<2.5 nm), anything greater than this may pose a problem and hinder one or more contacts and therefore give an erroneous measurement. In this case it is advised that the wafer is subjected to a Buffered HF dip for roughly 2 minutes to strip off any excessive oxide growth.

Trial	Site 1 (Rs)	Site 2 (Rs)	Site 3 (Rs)	Site 4 (Rs)
1	154.1812	154.5125	154.0188	152.8438

2	153.1563	154.0437	154.6000	153.8938
3	153.1750	153.9063	153.2937	153.2063
4	153.6938	154.4688	154.8188	153.2813
5	154.7312	155.1750	154.0375	154.0562
Mean (Rs)	153.7875	154.4212	154.1538	153.4563
Std Dev (%)	0.440%	0.322%	0.386%	0.329%

Table 2: Example of the measured sheet resistance output from a repeatability test performed by the 4D 4pp instrument on a silicon wafer, using the configuration shown below.

05/01/2010 – Configuration of 4pp test:
Probe Head Type : A
Test Radius : 15 mm
Offset : 1 degree
Layer Type : P
Gain Control : LOW
Geometric Correction : Off

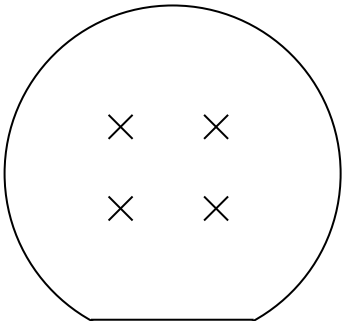


Figure 5: Sheet resistance repeatability test

It is also possible that even with an ideal substrate the probe tips refuse to form an ohmic contact which may mean the probes need reconditioning. After many measurements the probe tips can become smooth, and in fact the tip needs to be slightly rough to "bite" into the surface region of the substrate. Reconditioning of the probe head can be achieved by raising and lowering the head on to a ceramic plate. This also helps clean the head of any contamination picked up from previously measured samples.

An example of measurement error is demonstrated in Figure 6. Here it is possible to see that there are sharp changes in the measured sheet resistance (Rs) which only relate to single measurements (indicated via red arrows).

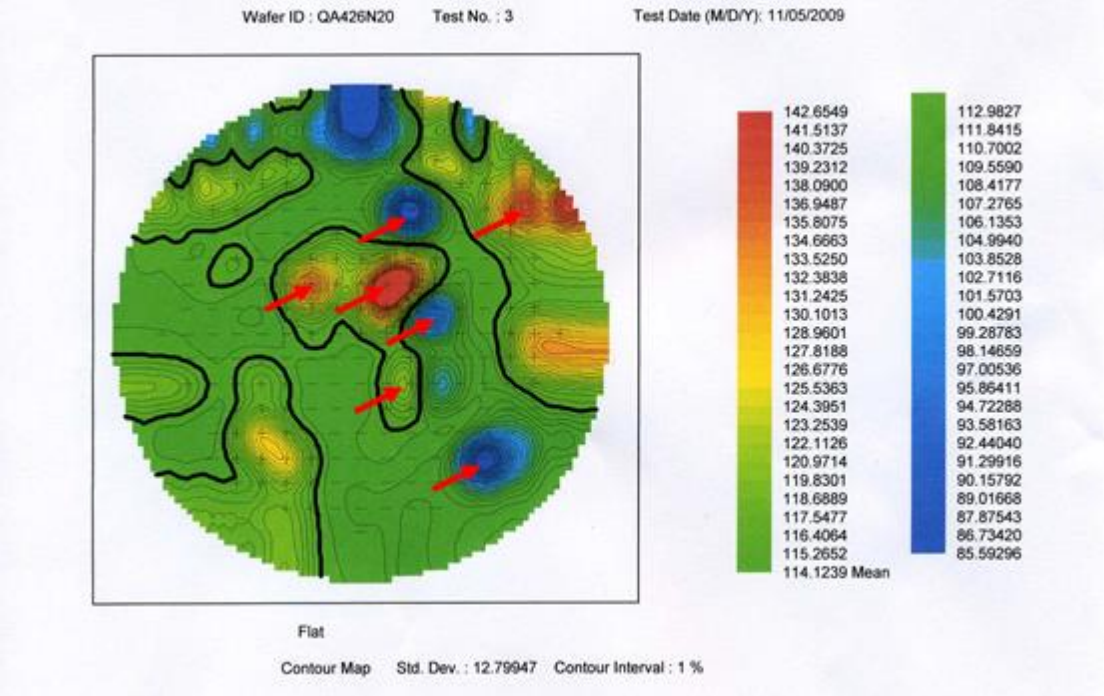


Figure 6: Typical 4pp map measured using a probe head whose probes need reconditioning.

If a similar effect is occurring on a measured wafer, the probe head may need cleaning/reconditioning. To do this the automated probe conditioning option does not work. Instead you have to simply make many single measurements under the diagnostic options to fool the system in to lowering the probe head onto the ceramic plate. 10 repetitions should suffice. If the problem persists the wafer may need cleaning and/or surface oxide stripping or the probe head needs replacing. If the repeatability is still above 0.5% after following the above procedure then the probe head needs to be replaced with a new one or one with re-conditioned probe tips.

5. Determining Anneal Schedules

The following section presents the results from a series of experiments which have been used to verify the anneal conditions for the Danfysik 1090 and the HV 2MV implanters at Surrey Ion Beam Centre and are used as a guide for others to determine their choice of schedule.

5.1 Danfysik 1090 - Boron

The standard Boron implant for the repeatability check on the Danfysik is performed at an energy of 30 keV and a dose of $1x10^{15}$ cm^{-2} down beam line 1. Boron is well known to form clusters with implantation damage below its solid solubility, therefore the anneal temperature

must be well above 800 ˚C to promote cluster dissolution. The SIMS analysis presented in Figure 7 illustrates the Boron profile before (dotted line) and after (solid line) annealing at 1000˚C for 3 minutes in a nitrogen atmosphere. It is possible to see that after annealing the peak of the Boron distribution lowers to roughly $3x10^{19}$ cm^{-2} which indicates that all the Boron Interstitial Clusters (BICs) have dissolved and due to the relatively high solubility limit it is possible to assume, indirectly, that all the Boron atoms are electrically active.

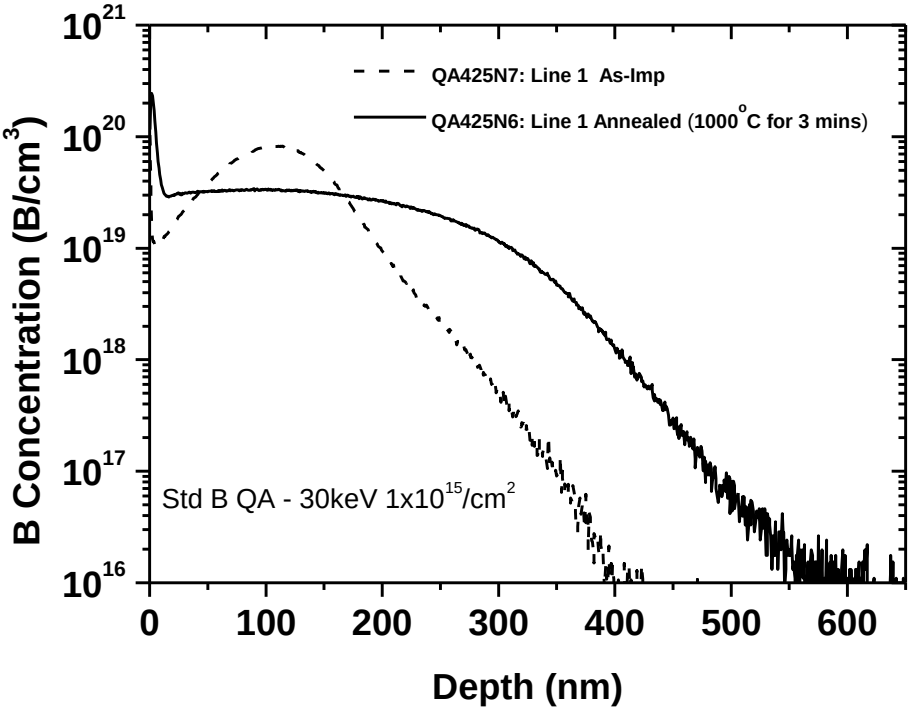


Figure 7: SIMS analysis before (dotted line) and after (solid line) a 1000˚C anneal for 3 minutes in a nitrogen atmosphere.

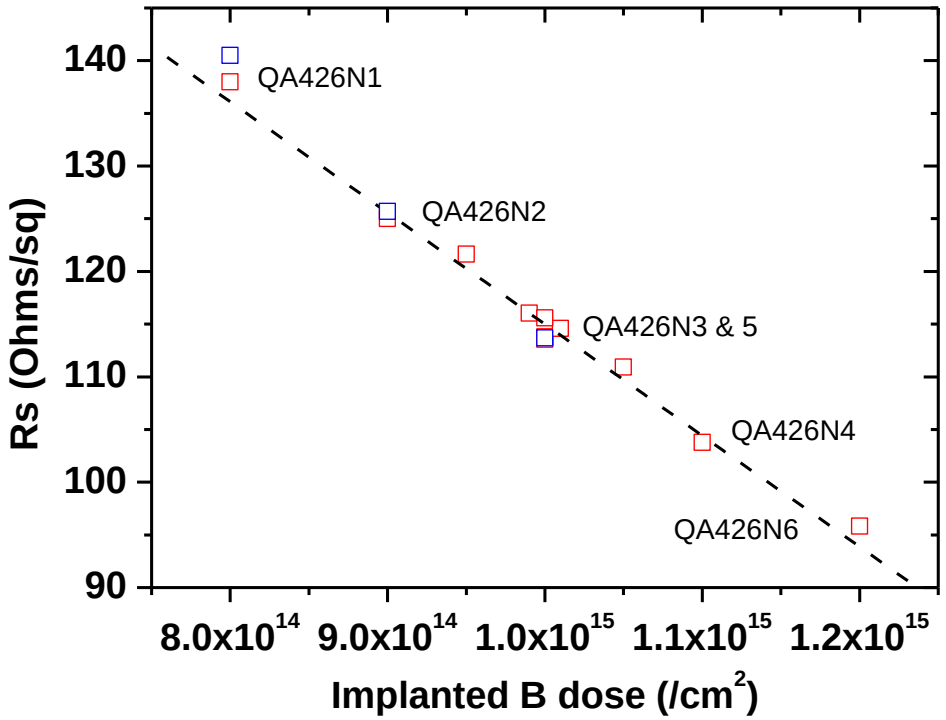


Figure 8: Mean sheet resistance (Rs) as a function of Boron implanted fluence (red squares) compared with simulations (blue squares) after a 1000˚C anneal for 3 minutes in a nitrogen atmosphere.

It is possible to compare actual sheet resistance (Rs) measurements with numerical simulations as performed in the modelling software SILVACO if the implant and anneal scheme are relatively simple. Figure 8 illustrates a series of actual Rs measurements as a function of implanted fluence (red squares) compared with simulations. All of the anneals were performed at 1000˚C for 3 minutes. As the simulations are in close agreement with the experimental values it is again possible to conclude that the Boron is 100% electrically active. The results also clearly show that it is possible to detect dose changes of the order of 1%.

5.2 Danfysik 1090 - Arsenic

The standard Arsenic implant is performed at an energy of 150 keV and a dose of $5x10^{14}$ cm^{-2}. It is possible to determine the substitutional fraction of an Arsenic implant after an anneal using RBS. However it is necessary to determine the sensitivity of implant to anneal temperature variations as this is a source of potential error. Figure 9 indicates that if the wafers

are annealed at 950 ˚C, even with a 25 ˚C error in temperature, the change in Rs is less than 1%.

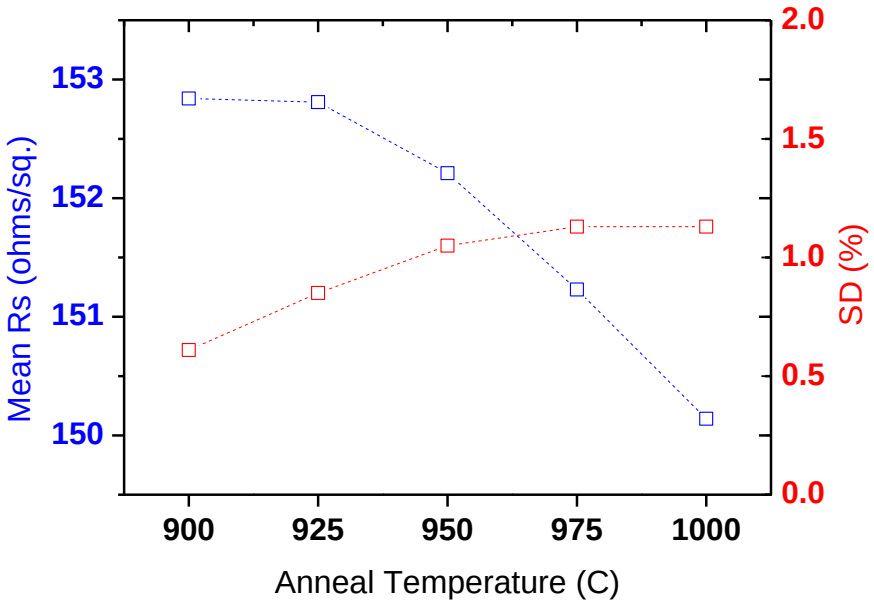


Figure 9: Mean sheet resistance versus anneal temperature for $5x10^{14}$ cm^{-2} Arsenic implant.

Figure 10 shows the sensitivity of the mean sheet resistance versus the nominal implanted dose around the QA implant dose of $5x10^{14}$ cm^{-2}.

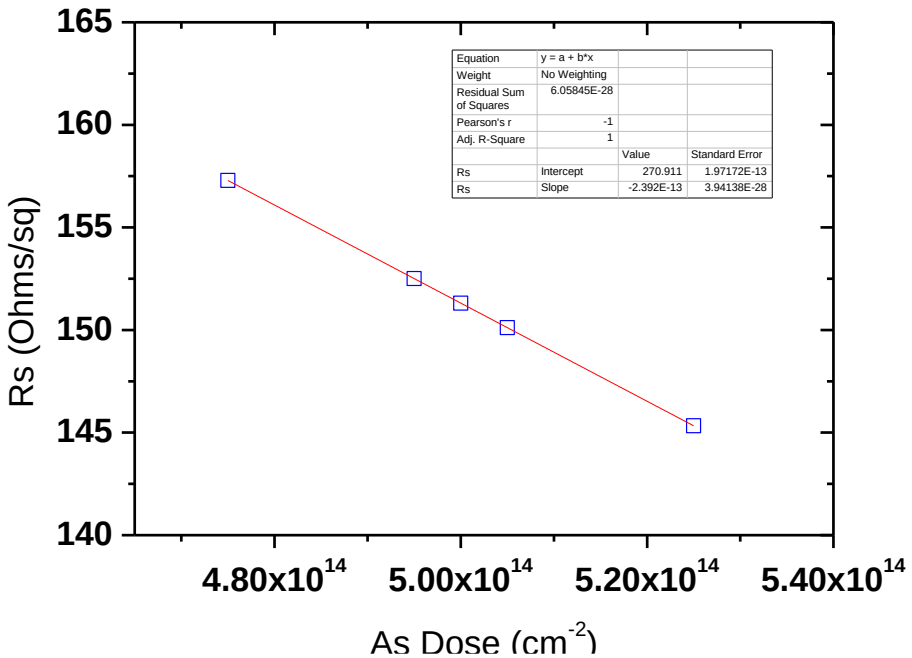


Figure 10: Mean sheet resistance versus Arsenic implant dose around $5x10^{14}$ cm^{-2} .

5.3 2MV – Antimony

For quality assurance of the higher energy 2MV implanter it was necessary to implant a heavier dopant atom than Boron or Arsenic as these elements are implanted too deep so that measurement of the sheet resistance with the 4D 4pp machine was problematic. Consequently a decision was made to implant Antimony instead. The standard Antimony implant used for wafer to wafer repeatability has an energy of 500 keV and a dose of $2x10^{14}$ cm^{-2}. It is also possible to determine the substitutional fraction of an Antimony implant after an anneal using RBS. In order to choose the most suitable implant for monitoring wafer repeatability, it was necessary to determine the sensitivity of the implant to both the dose and the anneal temperature. Figure 11 shows a plot of mean sheet resistance over a wide range of implanted dose. The graph indicates that the measured mean sheet resistance becomes less sensitive to the implanted dose as the dose increases. The dose of $2x10^{14}$ cm^{-2} was chosen for Antimony QA repeatability implants for its high dose sensitivity and repeatability of measurement.

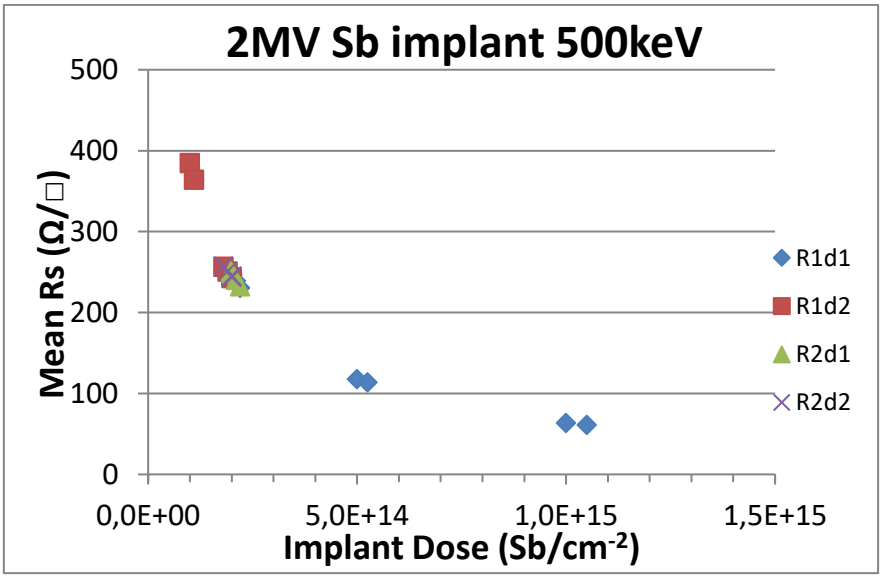


Figure 11: Mean sheet resistance versus the Antimony dose for a 500 keV implant on the 2MV VdG implanter.

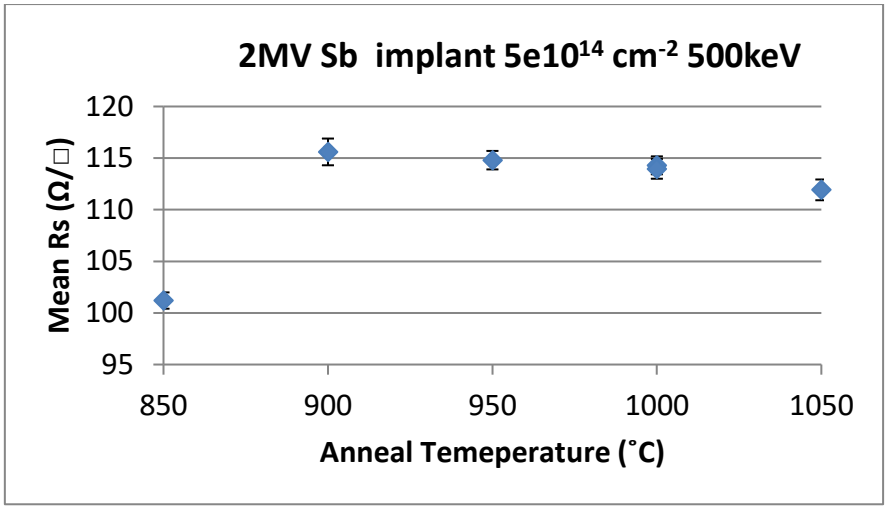


Figure 12: Mean sheet resistance versus anneal temperature for a 500 keV 5×10^{14} cm^{-2} Antimony implant on the 2MV VdG implanter.

Figure 12 shows the variation of the mean sheet resistance as a function of anneal temperature from 850 ˚C to 1050 ˚C. Figure 12 shows that the mean sheet resistance increases significantly from 850˚C to 900˚C and decreases markedly from 1000˚C to 1050˚C. An anneal temperature of 950˚C was chosen for Antimony QA wafer annealing so that the anneal is not sensitive to temperature variation but at a high enough temperature for activation of the implanted dopant.

6. Energy Calibration for High Energy Implanter

Using the gamma radiation generated when H^+ strikes an Al target it is possible to check the energy of the beam being supplied to better than 1% using the resonant capture reaction $^{27}Al(p,\gamma)^{28}Si$ at 992 keV. A calibrated gamma detector is placed as close as possible to the reaction point.

Other resonance points can be used for energy calibration and should be recorded at least annually.

7. RBS Analysis of Arsenic Implanted Wafers

A detailed explanation of Rutherford backscattering spectrometry (RBS) is outside the scope of this manual and can be found in the literature [ii]. This section presents an example of the data required to be recorded every quarter for QA purposes.

The majority of the time an acquisition of the random spectrum is sufficient. If the success of an anneal is in question then a channelled spectrum would need to be captured for comparison.

RBS is not only used to determine the absolute implanted fluence, but a minimum 5 point (up to 23) wafer map is also performed to ascertain implant non-uniformity across the wafer for comparison with the 4pp map. An example of the RBS measurement locations across a wafer can be seen in Figure 13.

RBS is usually performed with a 1.5 MeV $^{4}He^{+}$ beam. The beam current is typically a few nA and the nominal beam size is a few mm. The RBS signal is measured by two detectors at two different angles.

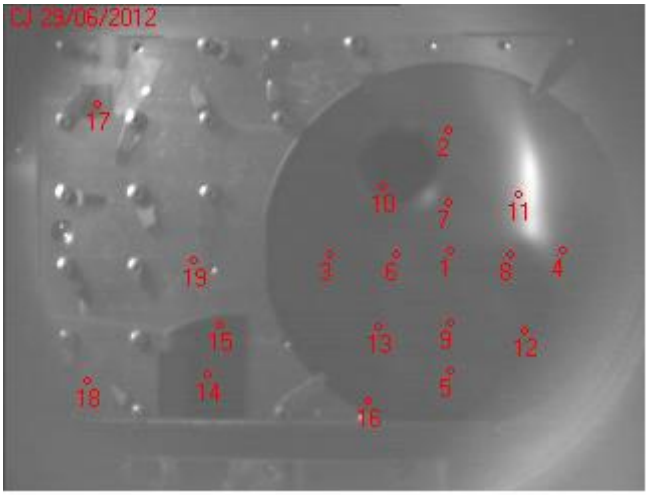


Figure 13: RBS typically measured at 13 locations across the As implanted QA wafer.

The calibration factor of the Tandem accelerator is routinely determined through the $^{16}O(\alpha,\alpha)^{16}O$ resonance at 3038 keV, as described in the literature [iii].

The exact gain and pulse height defect (PHD) of the electronics channel for each detector are determined through the spectra acquired on a reference sample. The PHD value of each detector is adjusted in order to get an offset close to zero, while the gain is left free to perform the fitting of the experimental spectra: the details are outside the scope of this Manual and can be found in the literature [iv] [v].

A typical depth profile from an Arsenic implanted QA wafer is shown in Figure 14. The RBS analysis concludes with the absolute measurement of the Arsenic fluence measured at all the locations across the wafer, an example of which is shown in Table 3.

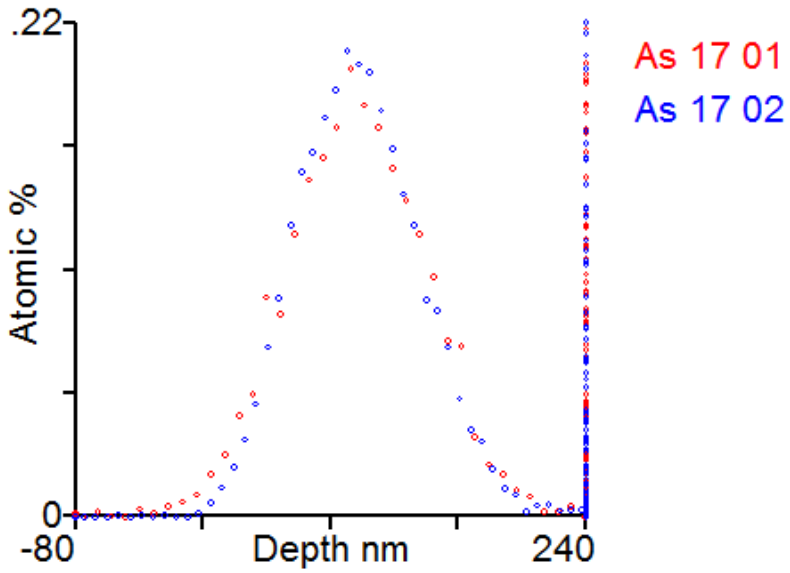


Figure 14: A typical depth profile from a 30keV $1x10^{15}$ Arsenic/cm^{-2} implanted silicon QA wafer for both detectors. These profiles are not resolution-corrected, but the areas under the two profiles are equal, within uncertainties.

Table 3 : As signal, 2 detectors					
Samples	Corrected DetA TFU	DetB TFU	Average TFU	Weighted Average TFU	DetA/ DetB
1	0.998	0.967	0.982	**0.975**	1.0320
2	1.058	1.090	1.074	**1.081**	0.9711
3	0.880	1.025	0.952	**0.985**	0.8584
4	1.123	0.943	1.033	**0.993**	1.1907
5	1.042	0.942	0.992	**0.969**	1.1067
6	1.008	0.954	0.981	**0.969**	1.0570
7	1.008	0.999	1.044	**1.023**	1.0896
8	0.988	0.969	0.979	**0.974**	1.0199
9	0.987	0.958	0.973	**0.966**	1.0301
10	1.007	1.067	1.037	**1.051**	0.9437
11	0.951	1.007	0.979	**0.991**	0.9448
12	0.955	0.987	0.971	**0.978**	0.9674
13	0.954	0.978	0.966	**0.971**	0.9757
Average	1.003	0.991	0.997	**0.994**	1.0144
ActualSD	6.40%	4.64%	3.71%	**3.58%**	8.41%
ExpectedSD	7059%	2.87%	2.68%	**2.68%**	8.11%
Real Variation	0.00%	3.65%	2.56%	**2.38%**	2.24%
Err in Average			1.03%	**1.03%**	2.33%
#measurements		13			
Uncertainty (coverage factor = 2)				**0.020**	
Combined uncertainty (coverage factor = 2)				**0.027**	

Table 3: Typical actual As fluence (measured by RBS) across a QA wafer (from QA Report).

[i] J. L. Colaux, C. Jeynes, K.C.Heasman, R.M.Gwilliam, Certified ion implantation fluence by high accuracy RBS, *Analyst* (in preparation)

[ii] C. Jeynes, N.P.Barradas, E. Szilágyi, Accurate determination of Quantity of Material in thin films by Rutherford backscattering spectrometry, *Analytical Chemistry* **84** (2012) 6061-6069

[iii] J.L.Colaux, G.Terwagne, C.Jeynes, On the voltage calibration of electrostatic accelerators, *Nuclear Instruments & Methods B* (in preparation)

[iv] J. L. Colaux, C. Jeynes, High accuracy traceable Rutherford backscattering spectrometry of ion implanted samples, *Analytical Methods* **6** (2014) 120-129

[v] J. L. Colaux and C. Jeynes, Accurate electronics calibration for particle backscattering spectrometry, *Analytical Methods* (in preparation)